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AN EVALUATION OF A GAS CHROMATOGRAPHIC METHOD FOR
THE DETERMINATION OF THE TRICHLOROFLUOROMETHANE
CONTENT IN THE STARTING POLYOL COMPONENT
OF POLYURETHANE FOAM

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PROPULSION AND VEHICLE ENGINEERING LABORATORY
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ABSTRACT

A gas chromatographic method for the determination of the trichlorofluoromethane content in a polyol has been evaluated. Certain modifications have been recommended. With these modifications, the method has been found to be satisfactory in the range of 28 percent \pm 2 percent trichlorofluoromethane, as required by Specification MB0130-077 (North American Rockwell Corporation material specification for two pound density polyurethane foam).

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SUMMARY

A gas chromatographic method for the determination of the trichlorofluoromethane content in a polyol has been evaluated. Certain modifications have been recommended. With these modifications, the method has been found to be satisfactory in the range of 28 percent \pm 2 percent trichlorofluoromethane, as required by North American Rockwell Corporation Specification MB0130-077.

INTRODUCTION

The North American Rockwell Corporation (the prime contractor for the S-II Stage of the Saturn V launch vehicle) has prepared a specification (MB0130-077) for the flame retardant polyurethane spray foam. The specification contains a chromatographic method for the analysis of trichlorofluoromethane content in the starting polyol component of the polyurethane foam. Since trichlorofluoromethane is used as a blowing agent for the polyurethane foam, it is mandatory that a chemical method be available to govern the correct concentration limits so that the correct foam density can be obtained. Trichlorofluoromethane is a highly volatile halogenated organic solvent that boils at 74°F. It is produced by various companies and marketed under trade names, some of which are as follows: DuPont - Freon 11, Allied Chemical - Genesolv A; and Union Carbon - Ucon 11.

This report covers the evaluation and modification of the chromatographic method in Specification MB0130-077 for the S-II program acceptability and for possible consideration of being incorporated into a spray foam material specification for MSFC.

Since the chromatographic method contained in the North American Rockwell specification appeared to be developed for use on a specific "home-made" apparatus, this report was prepared from an outlined program that produced an evaluation of the chromatographic method and also defines the instrumental parameters which would permit adaptation of the method to any commercially available gas chromatographic equipment.

EXPERIMENTAL

The initial review of the chromatographic method in Specification MB0130-077 (see appendix) showed that several vital pieces of information were missing such as carrier gas flow rate, inlet and detector temperatures, relative retention of trichlorofluoromethane, response factor for trichlorofluoromethane, the precision and accuracy of the method, and minor components of the polyol that may not resolve from the trichlorofluoromethane or the toluene. With recognition of these basic deficiencies, the evaluation was begun.

After a communication with personnel of North American Rockwell Corporation and after receiving a sample of the base polyol without trichlorofluoromethane, the equipment was set up to match as closely as possible the parameters of the proposed method (Table I).

As a beginning, samples of toluene and of trichlorofluoromethane were chromatographed to obtain some retention data on these two components. Next, an eight microliter ($8\ \mu\text{l}$) sample of the base polyol without trichlorofluoromethane was chromatographed to determine if there were any "light ends" which might elute with the same retention time as the toluene and/or the trichlorofluoromethane. No components were detected in 18 minutes after injection of the polyol; this was approximately 10 times the retention of toluene and trichlorofluoromethane.

Next, a calibration mixture of polyol, trichlorofluoromethane, and toluene was prepared in accordance with the proposed method. Over a two-day period, 23 replicate samples were injected and response factors were calculated to determine the reproducibility of the chromatographic method as written.

In addition, a study of the effect of inlet temperature on the detector response and on the shape of the toluene and the trichlorofluoromethane peaks was made. The inlet temperature was varied over the range of 100°C to 340°C holding all other parameters constant. Eleven Samples of the calibration mixtures were used in this study. Peak shapes were noted and the response factors were calculated for each injection.

Based on the work done to this point, the instrument was set up according to the parameters which would produce the best results from the method (Table II). Under these conditions, trichlorofluoromethane had a retention of 0.67 centimeters and toluene 4.05 centimeters. The relative retention (R/R) of trichlorofluoromethane was 0.409 (toluene = 1.00), where

$$R/R = \frac{\text{retention of Trichlorofluoromethane (cm)}}{\text{retention of Toluene (cm)}} .$$

A fresh calibration mixture and three samples of known trichlorofluoromethane content were prepared. Ten consecutive injections of the calibration mixture were made, and the response factor for each injection was calculated. The average value for the 10 calibrations was used in the calculation of trichlorofluoromethane content in the three known samples. Five or six injections of each sample were made, and the trichlorofluoromethane content was taken as the average of the individual injections of each sample.

As a word of caution, the operator must be certain that no air bubbles are present in the syringe when a sample is injected in the gas chromatograph. Large amounts of air will overlap the trichlorofluoromethane peak, giving an erroneously high value for trichlorofluoromethane content of the polyol sample.

The response factors and the trichlorofluoromethane content of the known samples were calculated by two methods. One method used peak heights measured in centimeters; the other used peak areas measured in square centimeters, where peak area = peak height x peak width at half-peak height. The data obtained by each method were compared for accuracy and precision.

In this work, the proposed method was followed as closely as possible, with one exception. All samples were made in 17-ml screw cap vials. After the polyol had been weighed into the vials, the vials were sealed in the following manner: A 1-inch square of 1-mil polyethylene film was placed over the mouth of the vial. Next, a 1/2-inch O.D. silicone rubber septum was inserted into the mouth of the vial. The septum was large enough to hold the polyethylene in place and proved to be a leak-tight seal. Finally, the rubber septum was held in place by a plastic screw cap into which a 1/4-inch O.D. hole had been drilled. The hole permitted injection of components into the vials and removal of samples for analysis.

Calculations

Peak Area = peak height (cm) x peak width at half-peak height (cm)

Relative Retention:

$$R/R = \frac{\text{retention of Trichlorofluoromethane (cm)}}{\text{retention of Toluene (cm)}}$$

Retention is measured on the chart paper from the point of sample injection to the maximum of the component peak in question.

Standard Deviation:

$$S = \sqrt{\frac{\sum (X_i - \bar{X})^2}{(n - 1)}}$$

where: X_i = each value in a series

\bar{X} = arithmetical mean of the series

n = the number of determinations.

Response Factor:

$$F = \frac{\text{Weight of Trichlorofluoromethane} \times \text{height of Toluene peak}}{\text{Weight of Toluene} \times \text{height of Trichlorofluoromethane peak}}$$

Trichlorofluoromethane Content:

$$\text{Wt. \% Trichlorofluoromethane} = \frac{\text{Weight of Toluene} \times \text{height of Trichlorofluoromethane}}{\text{Weight of Sample} \times \text{height of Toluene}} \times F \times 100$$

RESULTS

This investigation has established the fact that there are no components in the sample of polyol used during this evaluation (BX-250-A, Lot No. 02-1625-5) which would interfere with the trichlorofluoromethane or the toluene peak response.

As outlined, the procedure was not satisfactory when it was reproduced on standard commercial equipment. As seen in Table II, the average deviation of F , 0.80 ± 0.19 , was not bad; but the size and shape of the peaks varied over a wide range throughout the series of injections (Fig. 1). This indicated that perhaps the sample was not being vaporized rapidly enough to give consistently sharp, symmetrical peaks.

An increase in inlet temperature solved the problem of poor peak shape. (See Table IV and Fig. 2.) Along with improved peak shape, a smaller average deviation of F , 0.68 ± 0.03 , was obtained. Also, now the parameters more closely matched the ideal situation - inlet temperature higher than the analytical column, and the detector the hottest point in the system so that no eluted components will condense.

Now, with the instrumental parameters well defined (Table II), the investigation of the method was begun in earnest. Table V gives a brief summary of the precision and accuracy of the results obtained by the method as modified. Table VI is a resume of the raw data obtained in this study.

The data calculated using peak height measurements give much better values than the data based on peak areas. This is due to the trichlorofluoromethane peak which is so sharp that it is very difficult to measure the peak width (on the order of 0.10 cm) to any great degree of accuracy. The best method of peak measurement is by electronic integrator as indicated in the proposed method. However, every laboratory may not have such an integrator at its disposal; therefore, the method was evaluated using the two means of manually measuring the component peaks. Either means of manual measurement is adequate for the purpose for which the method is intended; however, peak heights should be used for greater confidence and accuracy.

The data calculated from peak height measurements is acceptable for determining the trichlorofluoromethane content by the gas chromatographic method as may be seen from the average and the standard deviations expressed in Table V.

CONCLUSIONS

In general, the gas chromatographic method for trichlorofluoromethane in polyol is acceptable as written. The idea of using toluene as an internal standard as well as a solvent, is a good one. The internal standard technique compensates for day-to-day variation in instrumental parameters and also permits variation in sample size without loss in accuracy or precision.

The data obtained during this study indicate that the method is acceptable for the analysis of trichlorofluoromethane content in the range of 28 percent \pm 2 percent as mentioned in Specification MB0130-077.

The instrument parameters listed in Table II are adequate for inclusion in a specification which requires a chromatographic method of analysis. If an electronic integrator is not available for measurement of peak areas, the peak height measurements can be used for calculating trichlorofluoromethane content.

This work has established that there are no components in the polyol which would interfere with the measurement of the toluene and trichlorofluoromethane peaks under the conditions listed in Table II. Also, the increase in inlet temperature has improved reproducibility greatly and there was no evidence of decomposition of the toluene or the trichlorofluoromethane at this elevated temperature.

TABLE I. PARAMETERS AS OUTLINED IN THE PROPOSED PROCEDURE

COLUMN	6' x 1/8" o.d. stainless steel packed with a coating of 5 percent Carbowax 20-M on 70-80 mesh Chromasorb G
CARRIER GAS	Helium at 15 ± 1 psig
COLUMN TEMPERATURE	150°C
INLET TEMPERATURE	150°C
DETECTOR TEMPERATURE	150°C
RECORDER	Bristol, 1 millivolt, equipped with a disc integrator, or equivalent
DETECTOR CURRENT	290 milliamperes

TABLE II. RECOMMENDED OPERATING PARAMETERS

INSTRUMENT	F&M Model 720, or equivalent
COLUMN	6' x 1/4" o.d. stainless steel packed with a coating of 10 percent Carbowax 20-M on Diatoport W (60/80 mesh)
CARRIER GAS	60 ml of helium/minute at 30 psig
COLUMN TEMPERATURE	150°C
INLET TEMPERATURE	220°C
DETECTOR TEMPERATURE	250°C
RECORDER	Honeywell, 0-1 mv, chart speed of 1 inch/min
DETECTOR CURRENT	150 milliamperes
SAMPLE SIZE	2 μ l from a 10 μ l syringe

TABLE III. PRECISION OF RESPONSE FACTOR CALCULATION USING
METHOD BEFORE MODIFICATION

Injection No.	Response Factor By Peak Height	Response Factor By Peak Area
1	0.66	1.67
2	0.64	1.65
3	0.56	1.36
4	0.73	1.71
5	1.18	1.69
6	0.69	1.89
7	1.58	1.46
8	0.65	1.67
9	0.65	1.72
10	0.68	2.08
11	1.58	1.78
12	0.69	1.94
13	0.66	2.12
14	0.67	1.90
15	0.77	1.79
16	1.11	1.73
17	0.68	2.06
18	0.71	2.13
19	0.80	1.86
20	0.73	2.03
21	0.70	1.81
22	0.58	2.15
23	0.72	1.76

TABLE IV. VARIATION OF RESPONSE FACTOR WITH INLET TEMPERATURE

INLET TEMPERATURE, °C	F _H	F _A
100	0.46	1.35
125	1.12	1.19
150	0.43	1.40
175	0.64	1.92
200	0.74	2.08
225	0.68	1.79
250	0.76	1.92
275	0.67	2.01
300	0.66	1.77
320	0.67	1.94
340	0.63	1.90

TABLE V. PRECISION AND ACCURACY OF TRICHLOROFLUOROMETHANE ANALYSES

Sample No.	% Trichlorofluoromethane Actual	% Trichlorofluoromethane by Peak Height	Standard Deviation	% Trichlorofluoromethane by Peak Area	Standard Deviation
1	27.95	27.35 ± 0.05	0.08	27.83 ± 0.47	0.83
2	31.02	30.92 ± 0.44	0.54	31.33 ± 1.04	1.45
3	33.08	32.69 ± 0.60	0.73	31.45 ± 1.41	1.74

TABLE VI. SUMMARY OF TRICHLOROFLUOROMETHANE ANALYSES OF
MODIFIED PROCEDURE

Sample No.	% Trichlorofluoromethane by Peak Height	% Trichlorofluoromethane by Peak Area
1	27.42	29.08
	27.35	27.94
	27.42	28.13
	27.22	27.75
	27.35	26.23
2	30.49	33.02
	31.47	32.23
	31.54	31.26
	30.28	31.06
	31.12	28.91
	30.63	30.28
3	32.94	30.67
	32.17	30.87
	33.92	33.60
	32.03	32.82
	32.38	29.30

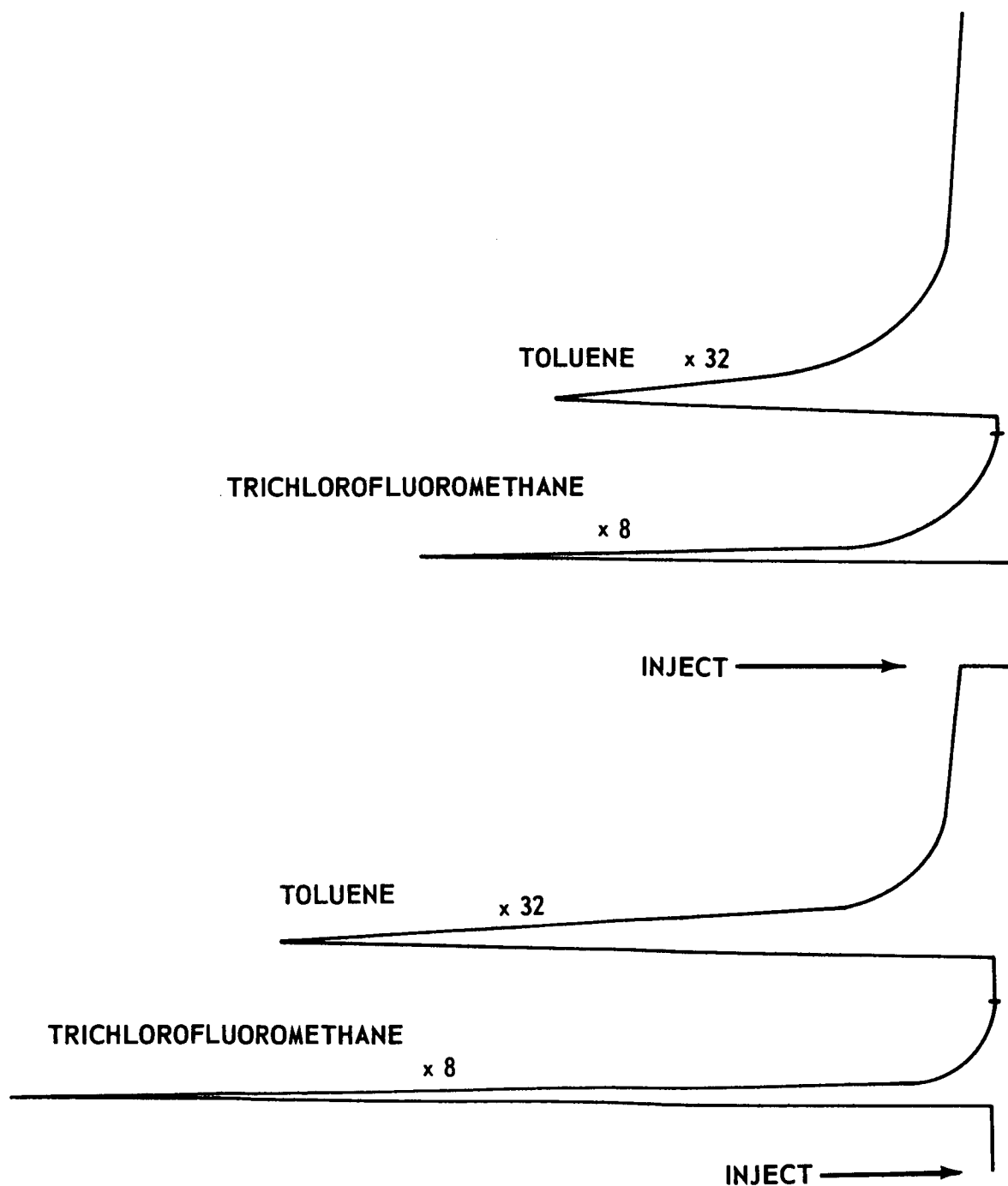


FIGURE 1 CONSECUTIVE 2- μ l INJECTIONS OF CALIBRATION MIXTURE

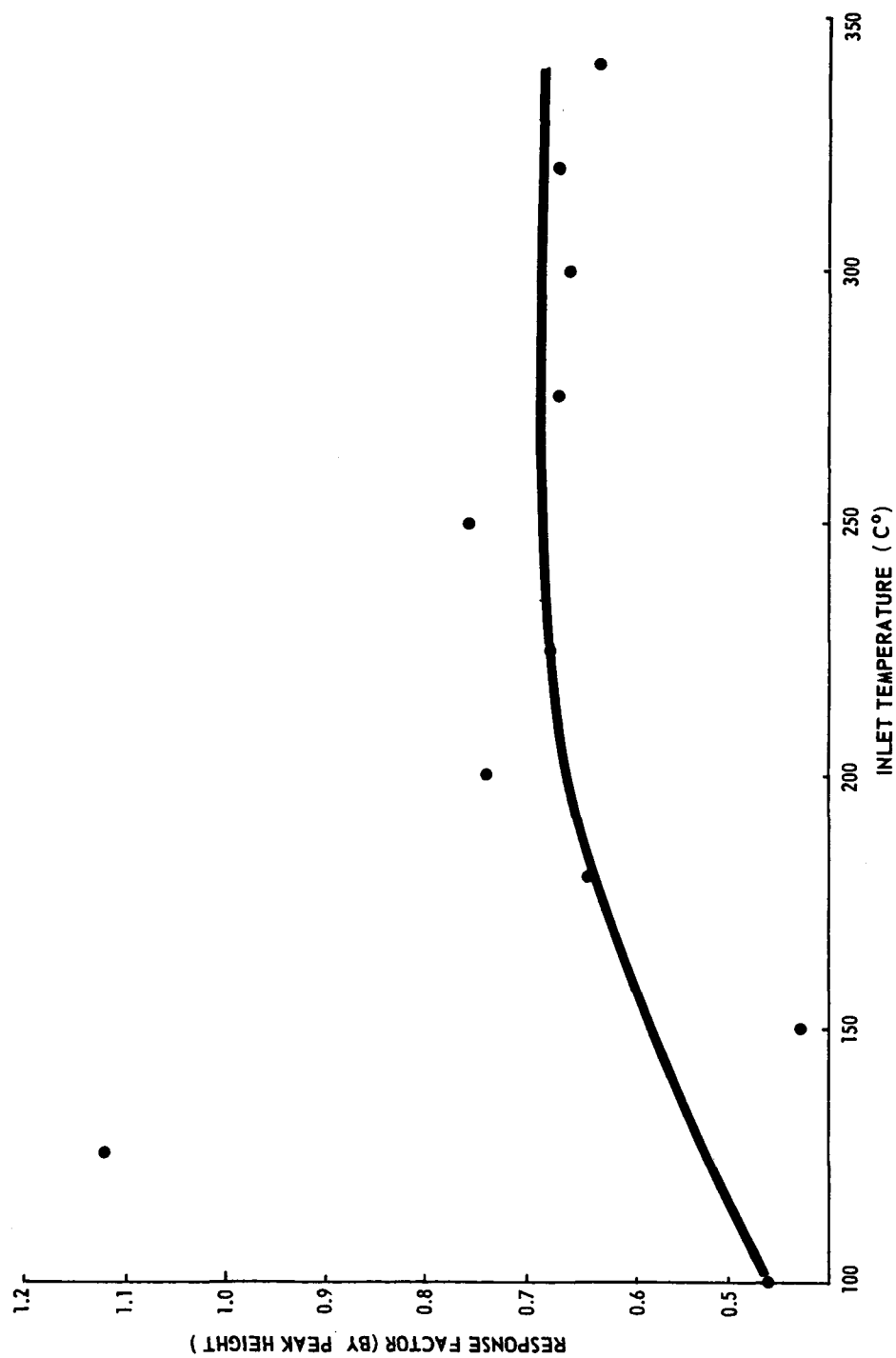


FIGURE 2 RESPONSE FACTOR VERSES INLET TEMPERATURE

REFERENCE

Specification MB0130-077: Flame Retardant Polyurethane Foam,
Two Pound Density, for Spray Application. Space Division, North
American Rockwell Corporation, Downey, California 90241.

APPENDIX

PROPOSED PROCEDURE FOR DETERMINING FLUOROCARBON CONTENT IN POLYOL*

1. Fluorocarbon Content

The percent fluorocarbon shall be determined by using one of the following procedures and shall meet the requirement in Table I.

a. Gas Chromatographic Method:

(1) Apparatus:

Gas Chromatographic System

Inlet, heated, Hamilton, Incorporated, Model 86800 with glass fiber packing in vaporizer tube, or equivalent.

Column, 1/8 in. o.d. x 6 ft. stainless steel, with 5 percent weight Carbowax 20M on 70-80 mesh Chrom G.

Detector, Gow Mac Model 9285 4-filament Hot Wire Thermoconductivity or equivalent, operated with 24 volt power supply.

Recorder, 1 millivolt, Bristol Model, equipped with a Disc Integrator, or equivalent.

Column compartment, temperature controlled at 302°F (150°C).

Serum bottles, 30 cc, with size E sleeve type stopper, or equivalent.

Syringe, 10 cc, gas tight, Hamilton Cat. No. 1010, or equivalent.

Syringe, 5 microliter, Hamilton Cat. No. 7005N, or equivalent.

* Excerpted from North American Rockwell Corporation Specification MB0130-077, "Flame Retardant Polyurethane Foam, Two Pound Density, for Spray Application."

(2) Calibration:

Assemble gas chromatographic system per Figure 1.

Supply helium gas to the carrier gas inlet at $15 \text{ psi} \pm 1 \text{ psi}$. Verify the gas flow by attaching one end of a length of tubing to the column exhaust and the other end in a beaker of water.

Preheat the gas chromatograph column, column compartment, and inlet to $302 \pm 9^\circ\text{F}$ ($150 \pm 5^\circ\text{C}$).

After the column has purged for a minimum of 30 minutes, adjust detector filament current to 290 milliamperes.

Turn the recorder on and adjust the attenuator and zero controls to put the recorder pen on the zero chart position.

An adequate baseline is achieved if the pen does not drift more than 1 percent full scale per minute.

Prepare the standard polyol by weighing a 30 cc serum bottle with a sleeve stopper.

Weigh a 4-5 gram sample of base polyol (component 'B' without the fluorocarbon blowing agent) into the serum bottle, seal the bottle with the sleeve stopper, and record polyol weight as 'A'.

Using the 10 cc syringe, inject a volume of reagent grade toluene (approximated by multiplying the base polyol weight by 1.6) into the serum bottle. Care should be taken:

- (a) to cover the rubber sleeve stopper with a sheet of polyethylene film to prevent the toluene from wetting the rubber stopper,
- (b) not to allow the syringe needle to come in contact with the base polyol in the serum bottle, and
- (c) to draw a volume of gas (equal to the volume of toluene added) from the serum bottle to equalize the pressure inside the serum bottle.

Re-weigh the serum bottle and record the weight of the toluene added as 'B'.

Using the following formula, calculate the approximate volume of Freon-11 blowing agent to inject into the serum bottle.

$$\text{Volume Freon-11 (cc)} = 0.3 \times 'A'$$

where:

A = weight of base polyol

Pre-cool the serum bottle, a clean 10 cc gas-tight syringe, and the Freon-11 blowing agent in a refrigerator (35-50°F) for 10-20 minutes.

Inject the volume of Freon-11 blowing agent into the serum bottle using the same syringe technique outlined for injecting the toluene.

Allow the serum bottle to warm to room temperature.

Re-weigh the serum bottle and record the weight of Freon-11 blowing agent added as 'C'.

Mix the solution thoroughly by shaking the serum bottle.

Using the 5 microliter positive displacement syringe, draw a 1 to 2 microliter sample into the syringe, adjust the plunger to deliver a 1 microliter sample, wipe any excess from the needle, inject the sample into the inlet of the gas chromatograph and adjust the attenuation as required to keep both the Freon-11 peak and the toluene peak on the chart.

Calculate the areas under both peaks using the integrator.

Calculate the Freon-11 factor and the percent Freon in the standard by using the following equation:

$$\text{Factor} = \frac{C}{B} \times \frac{D}{E}$$

$$\text{Percent Freon} = \frac{C \times 100}{A + C}$$

where:

A = weight of base polyol

B = weight of toluene

C = weight of Freon-11

D = area under the toluene peak

E = area under the Freon peak

Average Factor shall be obtained by repeating the one microliter sampling ten times and averaging the results.

Tare a 30 cc serum bottle and sleeve stopper.

Pre-cool the capped serum bottle and a dropping pipette for 10-20 minutes in a refrigerator.

Open the component 'B' container cautiously and quickly transfer a sample of the component 'B' (approximately 5 cc) to the serum bottle using a dropping pipette and seal immediately with the sleeve stopper. Do not allow the sample to contact the neck of the serum bottle.

Allow the serum bottle to warm to room temperature, reweigh the serum bottle, and record the weight of the component 'B' as 'F'.

Using the toluene injection method described under calibration, inject a volume of toluene (approximate volume is determined by multiplying the component 'B' weight by 1.25) into the serum bottle.

Reweight the serum bottle and record the toluene weight as 'G'.

Mix the sample thoroughly by shaking the serum bottle until a clear homogeneous solution is obtained.

Using the 5 microliter positive displacement syringe, inject a one microliter sample into the inlet using the procedure outlined under calibration and adjust the attenuation to keep the peaks on the chart.

Calculate the area under the peaks using the integrator.

Calculate the Freon content in the component 'B'; using the following equation:

$$\text{Percent Freon} = \frac{G \times H}{F \times I} \times \text{Factor} \times 100$$

Where:

F = weight of sample component 'B'

G = weight of toluene

H = area under Freon-11 peak

I = area under toluene peak.

Repeat the 1 microliter injections of the sample 5 times and average the Freon percentage for all 5 samples.

TABLE AI. FOAM COMPONENT PROPERTIES

Property	Requirement (1)		Test Paragraph
	Component A	Component B	
Viscosity, CPS (2)	275 ± 125	340 ± 100	4.3.1
Specific Gravity (2)	1.237 ± 0.006	1.208 ± 0.012	4.3.1
Amine Equivalent	134 ± 3	-	4.3.1
Water Content, Percent	-	0.1 maximum	4.3.1
Hydroxyl Number (3)	-	505 ± 20	4.3.16
Fluorocarbon Content, percent	-	28 ± 2	4.3.2
<p>(1) Minimum 4 samples per condition</p> <p>(2) Temperature shall be maintained at 73.0 ± 1°F.</p> <p>(3) To be determined on component 'B' prior to addition of the fluorocarbon blowing agent.</p>			

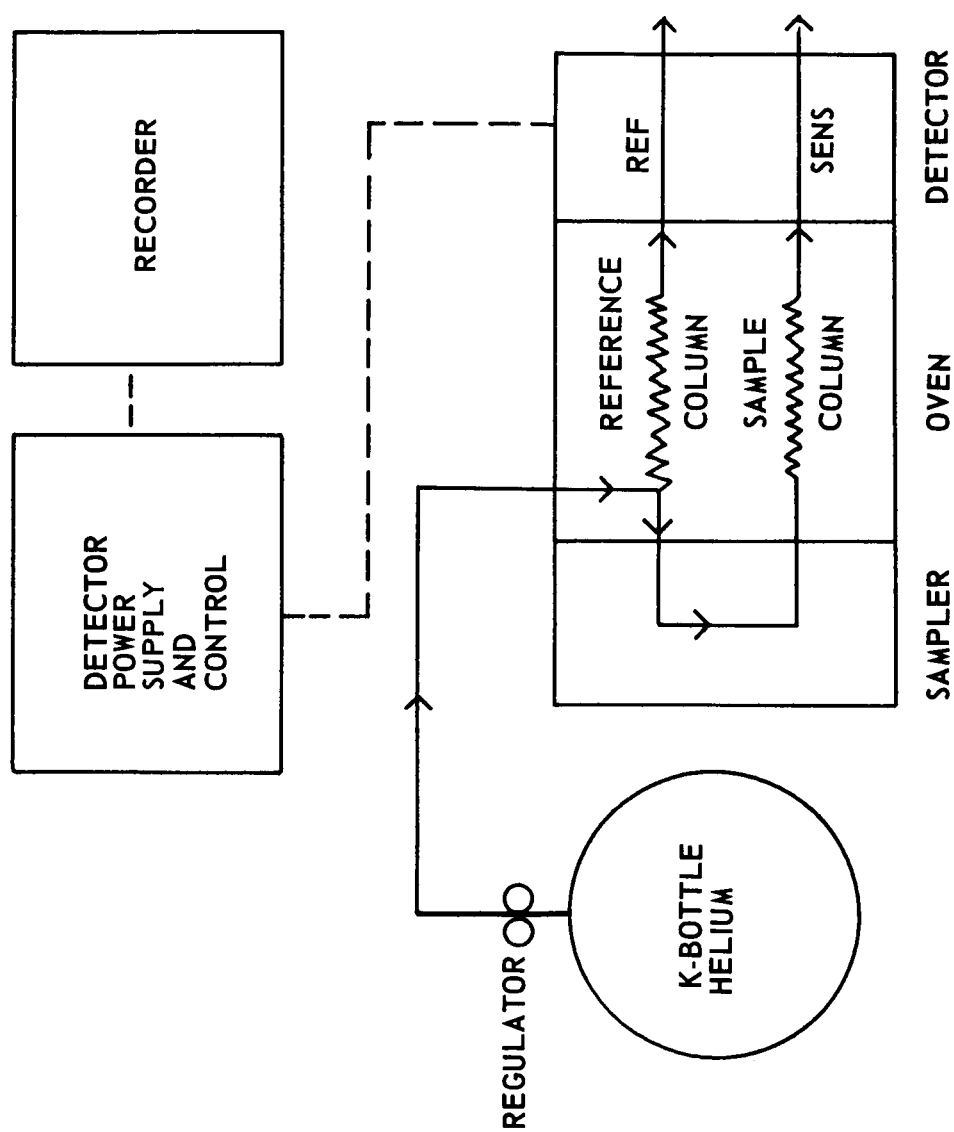


FIGURE A1 SIMPLIFIED DIAGRAM OF GAS CHROMATOGRAPH SYSTEM

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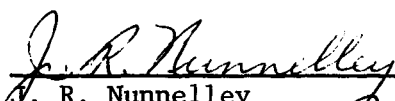
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The information in this report has been reviewed for security classification. Review of any information concerning Department of Defense or Atomic Energy Commission programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.

This document has also been reviewed and approved for technical accuracy.


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